By-product Iron-humate Increases Tree Growth and Fruit Production of Orange and Grapefruit

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Abstract. Citrus trees planted in alkaline soils typically show iron (Fe) deficiency chlorosis. Currently, Fe-EDDHA (ethylenediiminobis-2-hydroxyphenyl acetic acid) chelate is the most effective source of Fe for high pH soils. Iron humate (FeH), a by-product of the drinking water decolorization process, was compared with Fe-EDDHA for Fe deficiency correction on nonbearing 'Ambersweet' orange and 'Ruby Red' grapefruit *Citrus paradisi* Macf., and bearing 'Hamlin' orange *Citrus sinensis* and 'Flame' grapefruit trees, all on Swingle citrumelo rootstock, planted on high pH (>7.6) soils. Iron humate was applied under the tree canopy in spring at rates from 2 to 200 g Fe (nonbearing trees), or 22 to 352 g Fe (bearing trees) per tree per year. Application of FeH to nonbearing trees decreased twig dieback rating and increased flush growth, flush color rating, tree size, and leaf Fe concentration. Addition of urea or ammonium nitrate to FeH did not increase Fe availability. Iron amendments (22 g Fe per tree per year) increased fruit yield after the 1st year of application. Further increases in the rate of Fe, from 22 to 352 g Fe per tree per year as FeH, did not significantly increase tree growth, fruit yield, or fruit quality. This study demonstrated that FeH was an effective Fe source for citrus trees planted on alkaline soils.

Calcareous soils account for 21% to 42% of the total land area in some major Florida citrus-producing counties (Obreza et al., 1993). Iron deficiency is a widespread problem in alkaline soils (Alva, 1992; Chen and Barak, 1982; Korcak, 1987; Vose, 1982). Foliar Fe application is usually ineffective in correcting Fe chlorosis in plants because translocation of absorbed Fe to new growth is minimal (Martens and Westermann, 1991). Soil application of Fe is more effective than foliar application, especially if organic Fe chelates are used rather than inorganic Fe salts (Anderson, 1982; Mortvedt, 1982).

The effectiveness of Fe chelates is related to their stability at various soil pH levels

(Norvell, 1991). The stability of EDTA (ethylenedinitrilotetraacetic acid)-chelated Fe breaks down at about pH 6.5, so it is not an effective Fe source for soils with pH >6.5 (Norvell, 1991). EDDHA-chelated Fe is stable up to pH 9.0 (Norvell, 1991). Thus, Fe-EDDHA is the preferred Fe source to correct Fe deficiency in plants grown in high pH soils. However, Fe-EDDHA is expensive, limiting its use to high-value crops. In an effort to reduce costs and maximize economic return for Florida citrus, we evaluated the potential of an alternative source of Fe.

By-product materials from mining and industrial operations have been used to alleviate Fe chlorosis in crop plants with varying degrees of success (Martens and Westermann, 1991; Parkpian and Anderson, 1986). A new Fe by-product of drinking water decolorization using ferric sulfate, containing high Fe concentration, has recently become available. Iron is complexed with organic fractions (primarily humic and fulvic acids) in the water, and settles as an organically chelated material referred to as Fe humate (FeH). Characterization studies confirm the organic chelation property of this Fe source (Sartain and Varshovi, 1992, unpublished data). An increasing number of water treatment plants are adopting the use of ferric sulfate, instead of using aluminum sulfate. As a result, the by-product FeH is readily available and is considerably more economical than Fe-EDDHA. Since FeH is a

by-product of the drinking water decoloration process, it does not contain any toxic elements, and soil application is environmentally safe. The objectives of this study were to evaluate the effects of several application rates of FeH to high pH soils on 1) growth and leaf Fe concentrations of nonbearing citrus trees, and 2) leaf Fe concentrations and fruit yield and quality of grapefruit and oranges.

Materials and Methods

Field experiments were conducted on calcareous soils in four commercial citrus groves in south Florida. Citrus trees at all sites were budded on Swingle citrumelo (susceptible to Fe deficiency) rootstock. Site 1 was 9-year-old 'Hamlin' orange trees planted in Wabasso sand (sandy, siliceous, Hyperthermic, Alfic Haplaquods); site 2 was 7-year-old 'Flame' grapefruit trees planted in Oldsmar fine sand (sandy, siliceous, Hyperthermic Alfic Arenic Haplaquod); site 3 was 5-month-old 'Ambersweet' orange trees planted in Winder sand (fine-loamy, siliceous, Hyperthermic Typic Glossaqualf); and site 4 was 3-year-old 'Ruby Red' grapefruit trees planted in Hallandale sand (siliceous, Hyperthermic Typic Psammaquent). Site 1 was in DeSoto County, and sites 2, 3, and 4 were in St. Lucie County. Selected chemical properties of the soils in the four sites are given in Table 1.

Sites 1 and 2. Bearing trees. Citrus trees were planted on 90- to 120-cm-high raised beds to improve surface water drainage and provide adequate well-aerated soil for root growth. Two rows of trees were planted 7.5 m apart (4.5 m between trees within rows) on beds 9 m wide. During bed formation, the soil excavated to form the water furrow was placed on the bed; thus, the bed top was composed of subsoil from the water furrow deposited over the natural surface horizon. Each plot consisted of four uniform, adjacent trees within a row. The middle two trees were used for leaf and fruit sampling and fruit yield measurement.

The treatments at sites 1 and 2 included four sources of Fe: 1) Fe-EDDHA (LibFer SP, 6% Fe; Allied Colloids, Suffolk, Va.); 2) unmodified Fe humate (Un-FeH; 18% Fe), which is mechanically dewatered and dried Fe humate without any amendments or further processing; 3) urea-amended Fe humate (Ur-FeH), which is similar to Un-FeH, except that it contains urea at 100 kg·Mg⁻¹; 4) ammonium-modified Fe humate (Am-FeH), which is similar to Un-FeH except that it contains ammonium nitrate at 100 kg·Mg⁻¹; 5) Fe-EDTA, which was used only at site 1.

Each Fe source was applied (broadcast, under the canopy) at 22, 44, and 88 g Fe per tree per year in a single application in early spring (March) to coincide with the emergence of spring vegetative growth and bloom. The control received no Fe amendments. The FeH sources were also applied at 352 g Fe per tree per year to evaluate the possible adverse effect of application of this by-product at a high Fe rate on tree growth, fruit quality, and production. The experiment was conducted

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Table 1. Selected chemical properties of soil from four field experiment locations in Florida.

Sites	Soil pH	Extractable elements ² (mg·kg ⁻¹)								
		P	K	Ca	Mg	Na	Cu	Fe	Mn	Zn
1. 'Hamlin' orange	7.6	24	60	1355	183	ND ³	4	22	5	8
2. 'Flame' grapefruit	7.8	70	140	8500	170	39	29	100	31	30
3. 'Ambersweet' orange	7.4	3	63	1978	63	ND	ND	12	ND	ND
4. 'Ruby Red' grapefruit	8.0	4	235	6739	81	ND	ND	8	ND	ND

²Soil samples from sites 1 and 2 were extracted with Mehlich 3 solution for all elements; soil samples from sites 3 and 4 were extracted with Mehlich 3 solution for P, K, Ca, and Mg, and ammonium-bicarbonate/DTPA (diethylenetrinitriropentaacetic acid) solution for Fe.

³Not determined.

with five replications using a randomized complete-block design, and repeated over 3 years. The citrus groves were managed according to the routine cultural practices outlined by the Univ. of Florida, Institute of Food and Agricultural Sciences (Tucker et al., 1995).

Leaves from 5-month-old spring growth were sampled annually from the middle two trees (40 to 50 leaves) of each plot in August. The leaves were washed in a mild detergent solution by rubbing both sides with cheesecloth, followed by a rinse in distilled water, a 20-s rinse in 5% HCl, and several rinses in distilled water. The leaves were dried at 70 °C for 48 h and ground to pass a 40-mesh screen. A 0.5-g subsample of ground leaf tissue was ashed in a muffle furnace at 500 °C for 5 h, the ash was dissolved in 20 mL of 1 M HCl, and Fe concentration was determined using inductively coupled plasma emission spectroscopy (ICPES) (Plasma 40; Perkin Elmer, Norwalk, Conn.).

At fruit maturity, 30 fruits were sampled from the middle two trees of each plot to determine percentages of juice, Brix, acid, and soluble solids. Because the grapefruit was repeatedly spot-picked to meet fresh-market standards, on-tree fruit yield was estimated by using fruit counts per tree and mean mass per fruit. For the 'Hamlin' oranges, fruit from the middle two trees in the plot were harvested and the yield measured using the conventional field box (40.8 kg), and yield was calculated on a mass basis. During the first 2 years, soil samples were taken from all plots at 0- to 15- and 15- to 30-cm depths. The soil was air-dried and extracted with Mehlich 3 (M 3) extractant (Mehlich, 1984), and the Fe concentration measured using ICPES. The soil pH was measured in a 1 soil : 2 water suspension (mass : volume).

Sites 3 and 4. Nonbearing trees. Soil bed, configuration, and row spacing at sites 3 and 4 were similar to those at sites 1 and 2, but nonbearing trees were planted 3 m apart in the row. Since the area covered by the young tree roots was relatively small, the treatments were applied on a per tree basis, i.e., single tree plots.

The treatments included three rates of Am-FeH, two rates of Fe-EDDHA, and one nontreated control. The Am-FeH source was identical to that used at sites 1 and 2, but the Fe-EDDHA, although chemically identical, was obtained from a different manufacturer, (Sequestrene 138, 6% Fe; Ciba-Geigy, Greensboro, N.C.). At site 3 ('Ambersweet' orange), Am-FeH was applied at 2, 10, and 100 g Fe per tree, and Fe-EDDHA at 1 and 2 g Fe per tree.

At site 4 ('Ruby Red' grapefruit), Am-FeH was applied at 4, 20, and 200 g Fe per tree, and Fe-EDDHA at 2 and 4 g Fe per tree. Initial applications were made in May, with six replications of each treatment. The FeH was surface-applied and lightly mixed with the top 1 cm of soil to prevent rain from eroding it from the sloped bed. For convenience of application of small quantities of Fe-EDDHA, it was dissolved in 4 L of water and applied to a trench ≈5 cm deep around the dripline of each tree, and the trench was backfilled. The study was conducted over a 3-year period.

Tree growth response was used to evaluate the treatment effects. Leaf/twig flush growth ratings (FGR) were taken 4, 12, 18, and 24 months after the initial Fe application for the 'Ambersweet' trees, and 4 months after initial application for the 'Ruby Red' grapefruit trees. The rating method was 1 = no new flush to 10 = numerous new twigs. Leaf flush color ratings (FCR) were taken at the same time as FGR for 'Ambersweet', and 4, 12, and 24 months after the initial application for 'Ruby Red'. The rating method was 1 = very chlorotic (pale-yellow) to 10 = dark-green. Twig dieback ratings (TDR) were taken 12, 18, and 24 months after initial application for 'Ambersweet'. The rating scheme was 1 = 10% of new twigs had died back from the tip, 2 = 20% of new twigs had died back, etc. Trunk cross-sectional area, canopy diameter, and tree height measurements were taken at 12 and 24 months for 'Ambersweet' trees and at 4, 12, and 24 months for 'Ruby Red' trees.

Nutrient concentrations in 5-month-old leaves were measured at 6, 18, and 24 months after the initial Fe application at both sites using the methods described for sites 1 and 2. Soil was sampled at each site to a depth of 20 cm, the concentrations of Fe measured in an ammonium bicarbonate-DTPA (diethylenetrinitriropentaacetic acid) extract (Soltanpour, 1985). The concentration of other elements were measured using M 3 (Mehlich, 1984) extraction as described for sites 1 and 2.

Results and Discussion

Effects of Fe amendments on leaf mineral element concentrations. Iron application increased leaf Fe concentration significantly during all 3 years in the 'Hamlin' orange and 'Flame' grapefruit trees, but in only 1 out of 3 years for 'Ambersweet' and 'Ruby Red' grapefruit trees (Table 2). At the bearing tree sites, no significant differences in leaf Fe concentration were found between unmodified FeH and

that modified using urea or ammonium nitrate sources, indicating that Fe availability from FeH was not influenced by addition of either urea or ammonium nitrate. Leaf Fe concentration of 'Hamlin' orange trees that received Fe-EDDHA or Fe-EDTA was greater than that of trees that received FeH. Leaf Fe concentrations of 'Flame' and 'Ruby Red' grapefruit trees receiving Fe-EDDHA were greater than those of trees receiving FeH in only 1 of 3 years.

Leaf Fe concentrations of 'Hamlin' orange and 'Flame' grapefruit trees that received no Fe were in the deficient range for bearing trees (<35 mg·kg⁻¹; Tucker et al., 1995) all 3 years. Although applying Fe amendments increased leaf Fe levels, they remained in the low to optimum range (35 to 60 mg·kg⁻¹) throughout the study. Leaf concentration data for 'Hamlin' orange and 'Flame' grapefruit trees (Table 2) are for the 22 g Fe per tree per year treatment only. No significant increase in leaf Fe concentration was evident as Fe rates increased from 22 to 352 g Fe using FeH sources, or from 22 to 88 g Fe using the Fe-EDDHA source (data not presented). Thus, under severe Fe deficiency conditions, a single application of Fe amendment, regardless of rate, resulted in a significant but marginal increase in leaf Fe concentration.

A single, high-rate application of Fe amendment that is unincorporated may result in some movement of a portion of the material from the sloped bed. Soil Fe analysis at the bearing tree sites 30 d after Fe application showed no evidence of increased Fe, compared with unamended soil (data not shown). The Fe amendment sources and rates did not affect concentrations of N, P, K, Ca, Mg, and Cu in 5-month-old spring flush of trees at all sites and most elements were within normal ranges (Table 3). Therefore, Fe was probably the primary limiting element at all sites.

Growth of nonbearing trees. Applications of Am-FeH increased 'Ambersweet' flush growth rating (FGR) and flush color rating (FCR), decreased TDR, and increased tree size (Table 4). The trunk cross-sectional area showed the greatest response to FeH amendment. 'Ambersweet' tree height and trunk cross-sectional area were also increased by application of Fe-EDDHA. Although the treatments were applied in a similar fashion, 'Ruby Red' grapefruit trees showed no significant improvement in visual appearance or growth relative to controls (data not shown).

Fruit yield and juice quality of bearing trees. In the 1st year, the fruit yield response of 'Hamlin' orange to Fe amendments was neg-

Table 2. Effects of various Fe amendments and rates of application on Fe concentrations in 5-month-old leaves of four scion varieties on Swingle citrumelo rootstock grown in high pH soils.

Scion cultivar	Fe source	Fe rate (g Fe/tree/yr)	Concn Fe (mg·kg ⁻¹)		
			1993	1994	1995
Hamlin orange	No Fe	0	15 c ^z	18 c	21 c
	Un-FeH	22	26 bc	28 b	36 b
	Am-FeH	22	28 b	28 b	37 b
	Ur-FeH	22	25 bc	27 b	40 b
	Fe-EDDHA ^y	22	46 a	43 a	50 a
	Fe-EDTA	22	41 a	45 a	48 a
	LSD (<i>P</i> < 0.05)		11	6	5
Flame grapefruit	No Fe	0	15 c	23 b	20 b
	Un-FeH	22	27 b	30 a	36 a
	Am-FeH	22	26 b	33 a	37 a
	Ur-FeH	22	25 b	35 a	34 a
	Fe-EDDHA	22	35 a	32 a	38 a
	LSD (<i>P</i> < 0.05)		5	7	5
Ambersweet orange	No Fe	0	53 a	58 b	47 a
	Am-FeH	2	53 a	64 ab	45 a
	Am-FeH	10	53 a	74 a	44 a
	Am-FeH	100	49 a	64 ab	48 a
	Fe-EDDHA	1	61 a	71 ab	45 a
	Fe-EDDHA	2	52 a	65 ab	46 a
	LSD (<i>P</i> < 0.05)		12	15	9
Ruby Red grapefruit	No Fe	0	51 a	58 a	33 c
	Am-FeH	4	49 a	48 b	40 b
	Am-FeH	20	54 a	47 b	39 b
	Am-FeH	200	48 a	45 b	37 bc
	Fe-EDDHA	2	55 a	52 b	45 a
	Fe-EDDHA	4	48 a	49 b	40 b
	LSD (<i>P</i> < 0.05)		6	8	5

^zMean separation within years and cultivars by LSD, *P* < 0.05.

^yEDDHA = ethylenediiminobis-2-hydroxyphenyl acetic acid; EDTA = ethylenedinitrilotetraacetic acid.

Table 3. Concentrations of mineral elements in 5-month-old spring flush of control trees from four sites that received no Fe amendments.

Scion cultivar	Year	N	P	K	Ca	Mg	Cu
		(%)			(mg·kg ⁻¹)		
Hamlin orange	1993	2.42	0.12	0.52	3.59	0.23	16
	1994	2.84	0.15	0.87	3.48	0.34	14
	1995	2.55	0.13	0.91	3.81	0.37	15
Flame grapefruit	1993	2.37	0.15	1.31	3.28	0.23	28
	1994	2.55	0.17	1.55	2.87	0.25	48
	1995	2.46	0.11	1.43	3.49	0.28	93
Ambersweet orange	1992	3.11	0.41	4.07	2.45	0.24	35
	1993	3.26	0.22	3.24	2.62	0.26	36
	1994	2.97	0.23	2.62	2.57	0.31	68
Ruby Red grapefruit	1992	3.10	0.21	1.80	2.88	0.26	34
	1993	2.79	0.18	2.09	3.37	0.28	201
	1994	2.09	0.17	1.78	3.36	0.24	83

Table 4. Effects of Fe-humate and Fe-EDDHA^z treatments on 'Ambersweet' orange tree growth.

Fe source	Fe rate (g Fe/tree/yr)	Mean			Canopy diam (CD) (cm)			Tree ht (TH) (cm)			Trunk CSA ^y (mm ²)		
		FGR ^x	FCR ^x	TDR ^w	12 mo	24 mo	ΔCD	12 mo	24 mo	ΔTH	12 mo	24 mo	ΔCSA
No Fe	0	3.8 b	4.7 b	1.1 a	35 b	80 b	45 b	83 b	124 b	41 b	109 b	451 b	342 b
Am-FeH	2	5.8 a ^u	5.8 a	0.5 bc	47 a	111 a	64 ab	97 ab	150 a	53 ab	182 ab	747 a	565 ab
Am-FeH	10	6.1 a	6.4 a	0.4 bc	46 a	114 a	68 a	96 ab	155 a	59 a	209 a	875 a	666 a
Am-FeH	100	6.8 a	6.3 a	0.1 c	51 a	116 a	65 a	100 a	156 a	56 a	228 a	786 a	558 ab
Fe-EDDHA	1	5.4 a	5.5 ab	0.6 ab	43 ab	100 ab	57 ab	89 ab	144 ab	55 ab	179 ab	718 ab	539 ab
Fe-EDDHA	2	5.8 a	5.9 a	0.1 c	45 ab	96 ab	51 ab	102 a	153 a	51 ab	219 a	928 a	709 a
LSD (<i>P</i> < 0.05)		1.7	0.9	0.5	11	25	19	14	20	14	80	283	223

^zEDDHA = ethylenediiminobis-2-hydroxyphenyl acetic acid.

^xFlush growth rating: 1 = no new flush; 10 = numerous new twigs.

^yFlush color rating: 1 = very chlorotic (pale-yellow); 10 = dark-green.

^wTwig dieback rating: 1 = 10% of twigs have died back from tip, 2 = 20% of twigs, etc.

^uTrunk cross-sectional area, measured 15 cm above the bud union.

^vMean separation within columns by LSD, *P* < 0.05.

ligible regardless of source of Fe (Table 5). In the 2nd year, fruit yield increased by 7.0 to 8.4 Mg·ha⁻¹ in the trees receiving FeH, and 7.0 Mg·ha⁻¹ in the trees receiving Fe-EDDHA. In the 3rd year, fruit yield increases were 2.5 to 6.1 Mg·ha⁻¹ for FeH (depending on the source of FeH), and 3.2 Mg·ha⁻¹ for Fe-EDDHA relative to the controls.

For the 'Flame' grapefruit experiment, fruit yield data was not available for the 1st year. During the 2nd year, yield increased by 1.5 to 2.8 Mg·ha⁻¹ in response to FeH (depending on the source of FeH) and 1.5 Mg·ha⁻¹ in response to Fe-EDDHA (Table 5). In the 3rd year, yields were 1.3 to 3.3 Mg·ha⁻¹ (FeH treatments) and 1.0 Mg·ha⁻¹ (Fe-EDDHA treatment) above that of the control trees. The fruit yield response to application of FeH was greater than that to application of Fe-EDDHA. However, application of FeH had no significant effects on fruit size, juice quality, or soluble solids of either 'Hamlin' orange or 'Flame' grapefruit (data not shown).

These field tests of several FeH preparations on four calcareous south Florida citrus groves showed that FeH increased leaf Fe concentration, young tree growth, and fruit yield. In some experiments, the positive response to FeH was equivalent to that of Fe-EDDHA chelate, which is the standard but more expensive Fe source currently available for use on calcareous soils. Applying high FeH rates to citrus trees did not result in negative effects on tree growth and production, or in the soil chemical composition. The average price of commercial Fe-EDDHA products (containing 6% Fe) in the Florida market is ≈\$23.50 per kilogram vs. \$0.22 per kilogram for FeH (containing ≈16% Fe). Due to the high cost of Fe-EDDHA, this product needs to be applied on a tree basis, whereas FeH can be broadcast using commercial fertilizer spreaders. Therefore, both product cost and application costs are greater for Fe-EDDHA than for FeH. This study clearly demonstrated the effectiveness of FeH for correction of Fe deficiency in citrus. Accordingly, FeH is an economical Fe source for citrus. Large-scale long-term demonstration studies in commercial citrus groves using fertilizer broadcast equipment are necessary to develop FeH application rate recommendations on an area basis.

Table 5. Fruit yield of 'Hamlin' orange and 'Flame' grapefruit trees as influenced by Fe source.

Fe source	Fe rate (g Fe/tree/yr)	Fruit yield (Mg·ha ⁻¹)				
		Hamlin orange			Flame grapefruit	
		1993	1994	1995	1994	1995
No Fe	0	21.7 b	12.8 b	23.8 c	17.2 c	24.2 b
Un-FeH	22	22.1 b ^z	21.2 a	26.3 bc	20.0 a	27.5 a
Am-FeH	22	23.3 a	19.8 a	29.9 a	18.7 b	25.5 b
Ur-FeH	22	22.5 a	20.1 a	27.8 b	18.7 b	25.5 b
Fe-EDDHA ^y	22	23.1 a	19.8 a	27.0 b	18.7 b	25.2 b
Fe-EDTA	22	17.8 c	12.4 b	24.9 c	NA ^x	NA
LSD (<i>P</i> < 0.05)		1.1	2.3	2.1	1.3	1.5

^zMean separation within columns by LSD, *P* < 0.05.

^yEDDHA = ethylenediiminobis-2-hydroxyphenyl acetic acid; EDTA = ethylenedinitrilotetraacetic acid.

^xNA = not applicable, i.e., Fe-EDTA treatment was not included in 'Flame' grapefruit experiment.

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